U. S. Department of Agriculture, Forest Service

FOREST PRODUCTS LABORATORY

In cooperation with the University of Wisconsin

MADISON, WISCONSIN

MODIFIED CROSS AND BEVAN METHOD FOR DETERMINING CELLULOSE IN WOOD

(As Used At Forest Products Laboratory, Madison, Wis.)

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April, 1934

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Procedure

A weighed sample of approximately 2 grams of wood (60-80 mesh; Note 1) held in a tared alundum crucible (porosity R. A. 98) is extracted in a Soxhlet extractor with alcohol-benzene mixture (Note 2) for 6 hours and dried by suction. It is then washed with alcohol to remove all the benzene, and washed with water to remove the alcohol. The excess water is removed from the wet sample by suction (Note 3). The moist sample is transferred to a 250-cc. beaker by means of a pointed glass rod. Complete removal of the contents of the crucible is attained finally by holding the suction flask at an angle and with the suction applied, washing the particles of wood adhering to the wall, into the lowest point in the crucible. (The tedious process of complete removal of wood from the crucible may be avoided by placing the empty crucible in the chlorination apparatus and chlorinating it simultaneously with the sample.) The wood is uniformly distributed over the bottom of the beaker with a glass rod and the beaker placed in the chlorinating apparatus where it is cooled with running water (Note 4). Chlorine washed with water is now introduced at the rate of several bubbles per second. After 3 to 5 minutes' chlorination the beaker is removed, sulphur dioxide water added to stop the oxidizing action of excess chlorine on the sawdust, and the contents filtered. The residue is washed with water, returned to the beaker, and 100 cc. of a freshly prepared 2 percent sodium sulphite solution is added. (The crucible placed in a 100-cc. beaker should be digested with 25 cc. of 2 percent sodium sulphite solution.) The beaker is held in a boiling water bath for 30 minutes with occasional stirring, the contents filtered, washed with 250 cc. of hot distilled water, and again chlorinated. This operation is repeated until the chlorinated material fails to discolor the sodium sulphite solution. Coniferous woods require four to six chlorinations, hardwoods two to four chlorinations.

After the final digestion with sodium sulphite solution the cellulose fibers are filtered in a clean, tared (Note 5) alundum crucible and washed. The residue of cellulose remaining in the crucible used during the chlorination may be removed by use of a rubber policeman (Note 6). The cellulose is then returned to the beaker, 200 cc. of water added, and heated on the steam bath with occasional stirring for 2 hours (Note 7). The cellulose is filtered, washed successively with 100 cc. hot water,

50 cc. of 10 percent acetic acid (Note 8), 500 cc. of hot water, 50 cc. of 95 percent alcohol, and finally with 50 cc. of ether (Note 9). After removal of the ether (Note 10), the tared crucible and its contents are dried at 105° C. to constant weight in an air oven which usually requires 3 to 4 hours. The crucible is placed in a stoppered weighing bottle while cooling in the desiccator. It is then weighed (Note 11).

Notes

1. Wood is reduced to sawdust with a saw or a coarse rasp and ground in a Wiley mill or disc mill. The fraction of sawdust which passes a screen 60 meshes and is retained on a screen of 80 meshes to the inch offers fewer difficulties in manipulation than other fractions. The removal of lignin from fractions below 40 mesh becomes more difficult due to slower penetration of the larger particles by chlorine. Using sawdust finer than 100 mesh some cellulose passes through the crucible during washing and filtering.

2. A mixture of 67 volumes of benzene and 33 volumes of 95 percent alcohol is used.

3. To allow uniform penetration of chlorine the sample should be dried by suction until "fluffy" and the clumps can readily be broken up by a glass rod. Frequently after the second chlorination the material becomes gelatinous and consequently difficult to dry on the filter. This difficulty may be overcome by transferring the sample to the beaker, adding 200 cc. of distilled water and digesting on the steam bath for an hour. The substitution of an unused crucible after the second or third chlorination is probably a better method for elimination of the filtering difficulties.

4. The apparatus for the above method is described by A. W. Schorger, Ind. Eng. Chem. 9:561 (1917). If it is desirable to measure the amount of chlorine used the apparatus described by Bray is suitable. M. W. Bray, Ind. Eng. Chem. (anal. ed.) 1:40 (1929).

5. The crucible is weighed in a glass-stoppered weighing bottle. The substitution of a clean crucible renders filtering and washing much easier. Furthermore, the crucible used throughout the determination contains salts which are difficult to remove.

6. To obtain a snow-white product the cellulose may be bleached by adding 20 cc. of 0.10 percent potassium permanganate solution to the cellulose (after the final digestion with sodium sulphite and washing out of sodium sulphite); allow to stand 10 minutes and render colorless with sulphur dioxide water. It is then filtered, washed, returned to the beaker, 200 cc. of water added, and heated on the steam bath with occasional stirring for 2 hours. The remaining procedure for washing

and drying is the same as above. The bleaching may be omitted, since the yields of unbleached and bleached cellulose agree within a few eenths of 1 percent.

7. The digestion of cellulose with water and thorough washing with hot water is necessary to remove traces of acids which are persistently retained. If the cellulose is incompletely washed, as may happen if washing is performed in the crucible, it may darken during drying. Furthermore, the removal of salts and acids by means of the digestion in hot water will eliminate hydrolysis of the cellulose residue during drying in the oven. This is especially important if alpha cellulose determination is to be made on the Cross and Bevan cellulose as the hydrolysis during drying produces considerable quantities of water-soluble material.

8. Following any acid treatment of cellulose it is recommended that the final wash water be made slightly alkaline with ammonia to neutralize traces of adsorbed acid. This prevents extensive degradation during drying which is an important factor if the alpha-cellulose determination is to be made on the cellulose.

9. The alcohol and ether used in washing should be free of acids. Washing with alcohol and ether prevents the cellulose from drying to a hard mass by removing the water.

10. Explosions may occur unless the ether is removed from the sample before placing in the oven. The ether may be removed by leaving on the suction for 30 minutes or by the use of a steam oven.

11. The crucible should be kept in the bottle during weighing as cellulose takes up moisture from the air very rapidly.